SYNTHESIS OF ZINC OXIDE NANORODS: SEED LAYER ANNEALING EFFECT ON ELECTRICAL CONDUCTIVITY

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I would like to dedicate this thesis to my beloved parents, brothers, sisters, wife and my daughter for their endless support and encouragement.

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ABSTRACT

Orientation of zinc oxide nanorods has a vital role in the performance of nanoscaled-based optoelectronic devices and biosensing. In spite of a lot of research, there is no clear mechanism for the growth and orientation of ZnO nanorods during hydrothermal deposition. Beside the other properties of ZnO nanorods the electron transport properties and electron conduction are very important, in successful immobilization of various biomolecules in the field of biosensing. In current study the orientated ZnO nanorods were fabricated through hydrothermal method from a lowest possible precursor concentration. The precursors concentration was varied from 1 to 9 mM at constant deposition time (6 hours) and temperature (90˚ C), on annealed (500 ˚C) and un annealed gold seed layers. The mean square roughness of annealed and un annealed seed layers are 6.711 and 8.839 nm respectively. The morphological study of the prepared ZnO nanorods revealed a clear improvement in orientation of ZnO nanorods grown on annealed seed layer compared to un annealed seed layer. The structural characterization through XRD reveals that there is a clear peak (002) in all the concentrations on annealed seed layer. The elemental analysis showed the presence of zinc and oxygen in all concentration along with gold, which was detected from the glass substrate coated with gold. The FTIR analysis revealed very less number of bands for oriented ZnO nanorods compared to the un oriented nanorods. These analyses were confirmed by conductivity test conducting using four point probe, which shows the conductivity of oriented ZnO nanorods grown on annealed gold seed is very much higher (up to 90.909 S/m) than the un oriented ZnO nanorods (maximum value 11.574 S/m) grown on un annealed gold seed layer. From the results it is concluded that the seed layer roughness has a vital role in orientation of ZnO nanorods which has a good effect on the electrical conductivity of ZnO nanorods.

ABSTRAK

Orientasi zink oksida (ZnO) batang nano memainkan peranan penting berasaskan prestasi peranti optoelektronik dan nano pengesan bio. Penyelidikan kebanyakan tidak menunjuk mekanisma yang jelas untuk pertumbuhan dan orientasi ZnO batang nano semasa pemendapan hidroterma. Ppengangkutan elektron dan pengaliran elektron juga sangat penting dalam kejayaan imobilisasi terhadap biomolekul yang pelbagai di dalam bidang pengesan bio. Kajian semasa menunjukkan saiz ZnO batang nano yang direka melalui kaedah hidroterma daripada kepekatan prekursor yang terendah. Kepekatan prekursor adalah berbeza-beza daripada 1 sehingga 9 mM pada masa pemendapan yang berterusan (6 jam) dan suhu (90˚C), suhu pemanasan haba (500 ˚C) dan tidak dipanaskan lapisan benih emas. Purata kekasaran lapisan yang licin dan tidak licin adalah 6.711 nm dan 8.839 nm. Kajian morfologi daripada penghasilan ZnO batang nano menunjukkan peningkatan yang jelas pada permukaan yang berhaba berbanding dengan lapisan benih yang tidak berhaba. Pencirian struktur melalui XRD mendedahkan bahawa terdapat puncak yang jelas (002) di semua kepekatan pada lapisan benih yang berhaba. Analisis unsur menunjukkan kehadiran zink dan oksigen pada semua kepekatan bersama-sama dengan saduran emas. Manakala, analisis FTIR menunjukkan jumlah jalur yang sangat sedikit berorientasi ZnO batang nano berbanding batang nano yang tidak berorientasi. Analisis ini telah disahkan oleh ujian kebolehaliran yang dijalankan menggunakan prob empat titik, yang menunjukkan kebolehaliran berorientasikan ZnO batang nano yang bertambah pada suhu pemanasan hababenih emas adalah lebih tinggi (sehingga 90.909 S/m) daripada tidak berorientasi ZnO batang nano (nilai maksimum adalah 11.574 S/m) yang dan tidak dipanaskan lapisan benih emas. Daripada keputusan ini dapat disimpulkan bahawa benih yang mempunyai lapisan kasar memainkan peranan penting dalam orientasi ZnO batang nano, di mana ia mempunyai kesan baik terhadap kebolehaliran elektrik.

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LIST OF SYMBOLS AND ABBREVIATIONS

Å	-	Angstrom
hν	-	Photon energy
β	-	Temperature coefficient
μm	-	Micrometer
GaN	-	Gallium nitride
SiC	-	Silicon carbide
Gν	-	Gibb,s free energy
Si	-	Silicon
Gs	-	Surface energy
r *	-	Critical radius
k	-	Boltzmann constant
J	-	Rate of nucleation
Au	-	Gold
Ef	-	Formation energy
Cr	-	Chromium
meV	-	Mega electron volt
Gpa	-	Gaga Pascal
F	-	Mechanical force
P	-	Polarization
α	-	absorption co-efficient
λ	-	Wavelength
GaN	-	Gallium nitride
SiC	-	Silicon carbide
Si	-	Silicon
GOx	-	Glucose oxidase

σ	-	Resistivity
Ω	-	Ohm
Min	-	Deepest peak value
Max	-	Highest peak value
Rpv	-	Peak to valley value
Rq	-	Root mean square roughness
Rz	-	Ten point average
α	-	Absorption coefficient
W	-	Tungsten
WC	-	Tungsten carbide
Os	-	Osmium
Eg	-	Energy band gap
mM	-	Millimolar
SEM	-	scanning electron microscopy
FESEM	-	Field emission scanning electron microscopy
AFM	-	Atomic force microscopy
XRD	-	X-Ray diffraction
nm	-	Nanometer
PVD	-	Physical vapor deposition
CVD	-	Chemical vapor deposition
UV	-	Ultra-Violet
Eg	-	Energy band gape
MOCVD	-	Metal organic chemical vapor deposition
EDX	-	Energy dispersive x-rays spectroscopy
FTIR	-	Fourier transform infrared spectroscopy
HMT	-	Hexamethylenetetramine
VLS	-	Vapor liquid solid
RMS	-	Root mean square
US $	-	United states dollar
MBE	-	Molecular beam epitaxy
PZT	-	Lead Zarconium titanate
NRs	-	Nanorods

LIST OF PUBLICATIONS

[1] **Younas Iqbal**., Mohd Kamarulzaki Mustafa., Nur Liana Muhammad Rosdi. (2016) "Effect of orientation and configuration of ZnO nanorods on electrical conductivity prepared through hydrothermal method on suspended substrate" (Vol 8, No2) Journal of science and technology.

[2] MK Mustafa., **Y Iqbal.,** U Majeed., & MZ Sahdan. (2017). "Effect of precursor's concentration on structure and morphology of ZnO nanorods synthesized through hydrothermal method on gold surface" (Vol. 1788, No 1. 030120) AIP Conference Proceedings.

[3] **Younas Iqbal**., Mohd Kamarulzaki Mustafa., and Mohd Zainizan Sahdan. (2017) "Synthesis, growth mechanism and analysis of solid ZnO nanospheres by hydrothermal process"Journal of Engineering and Applied Sciences, Medwell Online.

[4] **Younas Iqbal**., M.K Mustafa., Mohd Zainizan Sahdan. (2017) "Orientation and the actual growth mechanism of ZnO nanorods through hydrothermal method at various precursor's concentrations" (Under review).

CHAPTER 1

INTRODUCTION

1.1 Nanotechnology and ZnO nanostructures

In 1959 Richard Feynman of California institute of technology delivered a lecture what is considered to be the first lecture on technology and engineering at the atomic scale. In his lecture he mentioned that "there is plenty of room at the bottom" suggesting manipulating things at the atomic level (Feynman, 1960). Feynman pointed out that there was a real possibility to design materials by manipulating individual atoms, as it would not violate any physical laws.

Nowadays this lecture is referred to as the origin of nanotechnology paradigm. However, because of the restriction in equipment capability, the boost of nanotechnology was observed in 1900s. In this decade, the, sophisticated equipment's such as scanning electron microscope, X-ray diffraction, atomic force microscopy and etc. have become more available for scientists to explore nanoworld. The word "nanotechnology" was first introduced by N. Taniguchi in 1974 at an international conference on industrial production in Tokyo in order to describe the super thin processing of materials with nanometer accuracy and the creation of nanosize mechanism. In the second half of 1980s and the early 1990s important discoveries and inventions were made which created an essential impact on the further development of

nanotechnology. Since then a number of publications on nanotechnological subjects and practical application of nanotechnology expanded sharply.

Fundamentally nanotechnology is the study of producing and operation of typical functional materials, devices and systems that fall in the range of 1-100 nm (Wang, 2005). In other words nanotechnology is a field of applied science that deals with synthesis, designing, characterization and application of nanoscale devices and materials (Abeer, 2012). Materials show a remarkable change in their fundamental properties (electrical and thermal conductivities, absorbance, photoluminescence, mechanical etc) when they are engineered at the nanoscale (Dubbaka, 2008). This is because of two major influences (i) high surface to volume ratio and (ii) quantum effects. High surface to volume ratio means that there will be a significant friction of atoms at the surface of the materials, which affects the electrical and mechanical properties, and high surface reactivity (Sirelkhatim, 2015), whereas the quantum effect is the confinement of charge carrier motion in small volume of material which affects the optical, electrical, magnetic, and thermoelectric properties of the materials (Gabrielyan, 2013). For example, opaque substance becomes transparent (copper); insulators become conductors; solids turn into liquids at room temperature (gold); and stable material turns into unstable materials (aluminum) (Yu, 2009). The electronic and optical properties of nanomaterials could be modified by modifying the charge carrier density and band structure in different way from their bulk counterpart. Nanoscale designing varies material's macro and microscopic properties such as charge capacity, magnetization, and melting temperature with unchanged chemical composition (Mathews, 1978). This means that as the particle size decrease the number of active atoms on the surface is significantly increase as shown in Table 1.1 (Chang, 2013; Hostetler, 1998).

In recent years a tremendous amount of research efforts has gone towards studying synthesis, fundamental properties, manipulation, characterization, device fabrication and testing. These new materials have a vital role and applications in industry sectors including medicines, security, transport energy and communication.

In year 2006, an estimated US $50 billion worldwide were amalgamated for nanotechnology, and was expected to reach US$1-2.6 trillion until 2011-2016. It is projected that the production of nanoparticles will increase from the estimated 2,300 tons today to 58,000 tons by 2020. Therefore, it is believed that nanotechnology will ultimately affect what the people eat, how they communicate, how long they live and

how they work. It will change their energy sources, medical care, water and environment (Schaefer, 2010).

Table 1.1: Comparison of percentage of surface atoms for Au nanoparticles

Particle radius (nm)	Total number of atoms	Percentage of surface atoms
0.65	79	76
0.71	116	67
0.81	140	69
0.87	201	64
0.98	225	62
1.10	309	52
1.20	459	51

Nanotechnology is considered to be the next industrial revolution and will cause tremendous impacts on the society, economy, and generally in future. It has wide applications in biotechnology, medicine, information technology, material technology, manufacturing, energy production and storage, instrumentation, environmental applications and security (Aneesh & Jayaraj, 2010).

In the world of nanotechnology, there are two schemes for making nanostructures and devices: Top-down approach and bottom-up approach. In top-down approach a bulk piece of material is divided and subdivided until it finally falls in the nanometer regime, whereas in bottom-up approach is building of a material from the bottom, i.e self-assembly of atoms and molecules into nanostructure: atom-by-atom, molecule-by-molecule or cluster-by cluster. The schematic of top down and bottom up approaches are shown in Figure 1.1. These various techniques are summarized in Table 1.2 (Khan, 2012). By comparing bottom-up and top-down techniques, the bottom-up techniques are far preferable as they exceed the limits of top-down technology regarding future applications, therefore the bottom up approach should be utilized as it is capable of producing nanoscale features (Wan, 2011). The other superiority of bottom-up approach over the top down is in terms of photolithography and is capable of producing variety of nanostructures with high yield, less defect and better range ordering, and is the reason that ZnO nanostructures fabricated through bottom-up approach possessed unique physical, electrical and optical properties which are highly applicable for downstream applications (Perumal, 2016).

Table 1.2: Summary of the various deposition and synthesis techniques to produce nanomaterials

Serial No.	General techniques	Sub techniques
1	Physical vapor deposition (PVD) methods	Magnetron sputtering Thermal evaporation Pulse laser deposition Radio-frequency Resistive Focused ion beam RF induction Electron beam Sputtering
2	Chemical vapor deposition (CVD) methods	Plasma-enhanced CVD Thermal CVD Metal-organic CVD Low-pressure CVD Molecular Beam Epitaxy (MBE) Atomic layer deposition
3	Solution based synthesis	Sonochemical method Sol-gel chemical process Hydrothermal/solvothermal method Micro-emulsion method Co-precipitation Template-assisted synthesis
4	Electrochemical synthesis	Electrochemical deposition Electrophoretic deposition
5	Physical methods	High energy ball milling process

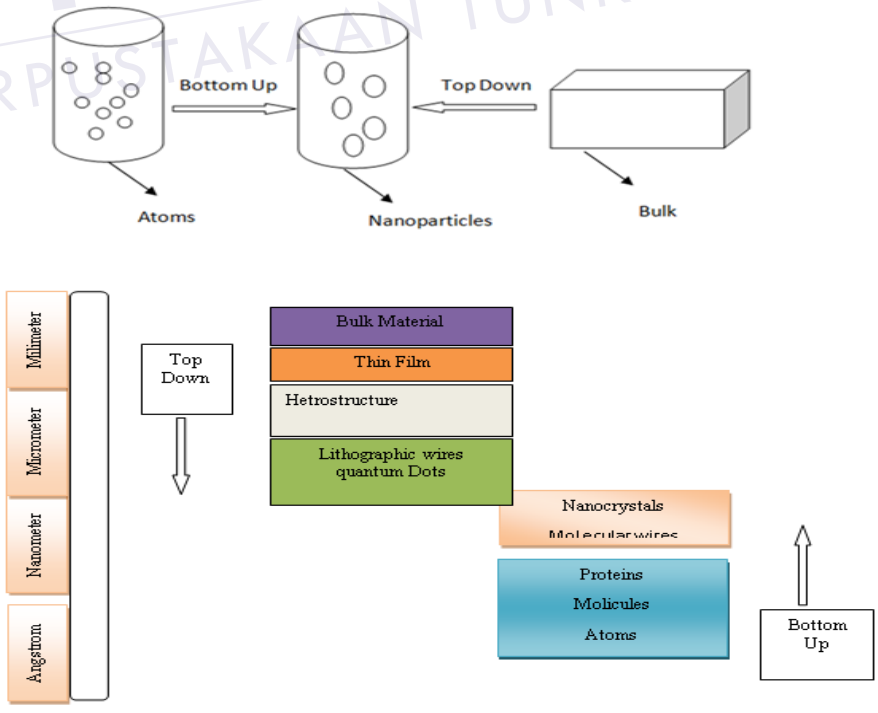

Figure 1.1: Schematic of top down and bottom up approaches

1.2 Characteristics and crystal structures of Zinc Oxide (ZnO)

ZnO is a wide direct band gap (3.37 eV) and a large exciton binding energy (60meV) semiconducting material at room temperature (Zhang., 2012). Materials are considered to be wide band gaps if their band gap energy is incomparably greater than that of common semiconductors like silicon (1.1 eV), and gallium arsenide (1.4 eV) (Yoder, 1996). The advantage of wide band gap material is that they are more efficient in high power and high temperature applications and can withstand high power because the electric breakdown voltage for wide band gap materials tends to be much higher (Rahmani, 2010). Furthermore, the large exciton binding energy (60 meV) than the thermal energy (25meV) at ambient temperature is responsible for stable electron-hole pair recombination, which paves a good way for the good luminescence behavior of ZnO (Lv et al., 2007).

Due to the large difference in electronegativity value (Zn=1.65, O= 3.44) the bonding between Zn atoms and O atoms is highly ionic (Jagadish & Pearton, 2011). ZnO belongs to II-VI semiconducting-materials and is found in two crystalline forms i.e hexagonal wurtzite and face centered cubic. In stable state ZnO usually exists in wurtzite while it can be turned into the zinc blend and rock salt phase by applying appropriate growth conditions (Alghamdi & Alzahrani, 2013). The rock salt phase of ZnO gives a smaller equilibrium volume than wurtzite phase because of the more tightly packed atoms in rock salt phase. In these crystalline structures, each anion is surrounded by cations at the corners of tetrahedrons. Each oxygen anion is surrounded by four zinc cations at the corner of the tetrahedron, and vice versa (Carpenter et al., 2012). There is a case when ZnO crystallizes in rocksalt structure when exposed to high pressure condition. Under compression at high pressure relatively 5-7Gpa, the lattice parameters reduce due to the interionic coulombic interaction to favor the iconicity more over the covalent nature and the wurtzite crystal structure transfer to the rock-salt type structure (Solozhenko et al., 2011). The rocksalt structure cannot be stabilized by the epitaxial growth (Morkoç, 2008). The zinc blende structure has lower iconicity as compared to the wurtzite structure and leads to lower carrier scattering and higher doping efficiencies (Ashrafi, 2007). Wurtzite is the more stable structure than rocksalt and zincblende, and it has been found theoretically that this crystal structure

is energetically more favorable in comparison to the other two structures. All these structures are shown in Figure 1.2.

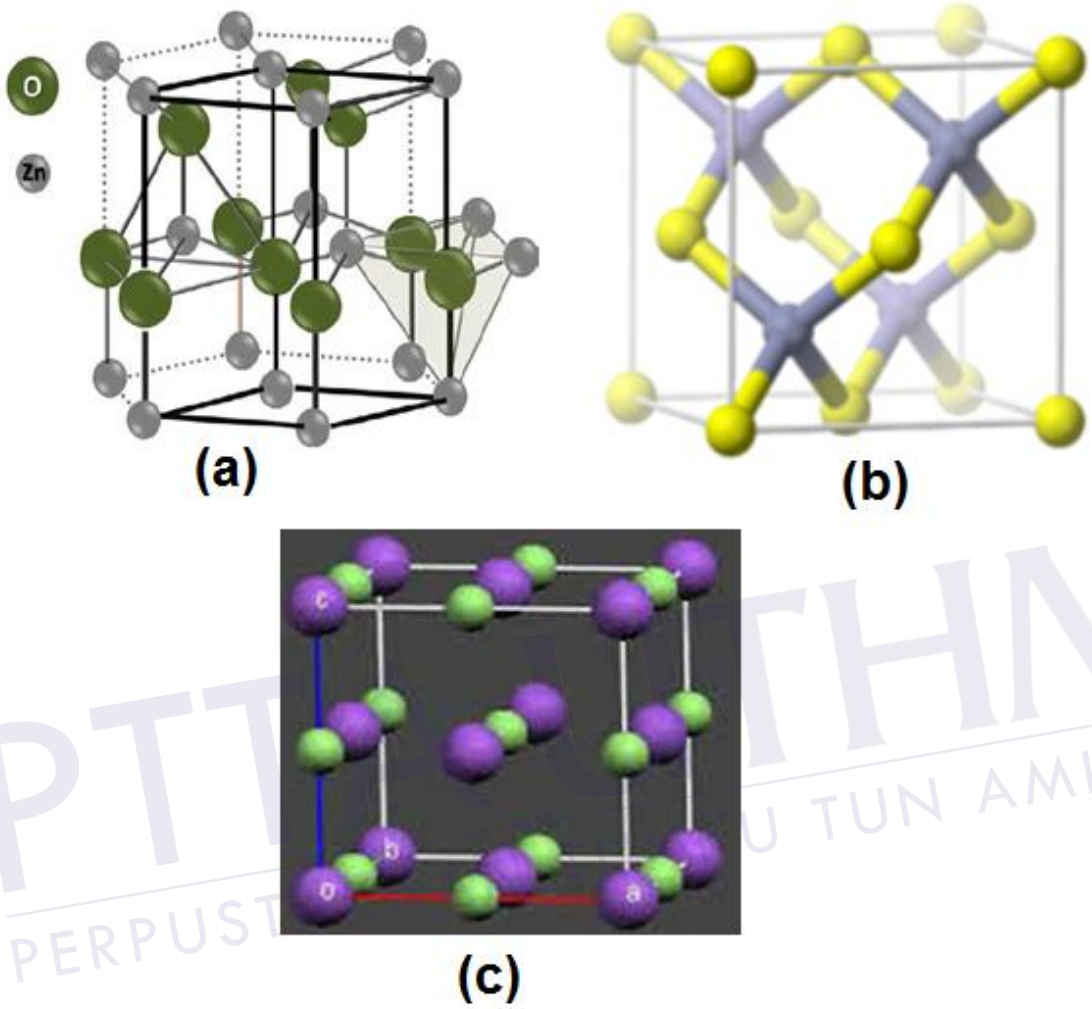

Figure 1.2: Crystalline forms of ZnO (a) Hexagonal wurtzite (b) Zinc blend (c) Rock salt (Ashrafi, 2007)

The structure of ZnO consists of alternating planes composed of tetra hedrally coordinated O-2 and Zn+2 stacked alternately along C-axis (Wang, 2004). Its structure is formed by alternating planes of tetra hedrally coordinated O-2 and Zn+2 ions throng along the c-axis as shown in Figure 1.3, which makes the entire structure without central symmetry, which result in a piezoelectric effect by which a mechanical stress/strain can be converted into electrical voltage, owing to the associative displacement of cations and anions in the crystal (Wang, 2008). Due to the specific distribution of cation (Zn2+) and anions (O2-) the entire unit cell of ZnO is neutral,

some surfaces are terminated entirely with cations or anions (Bing et al., 2014), which leads ZnO possesses positively or negatively charged on the surface (Birkholz, 2006).

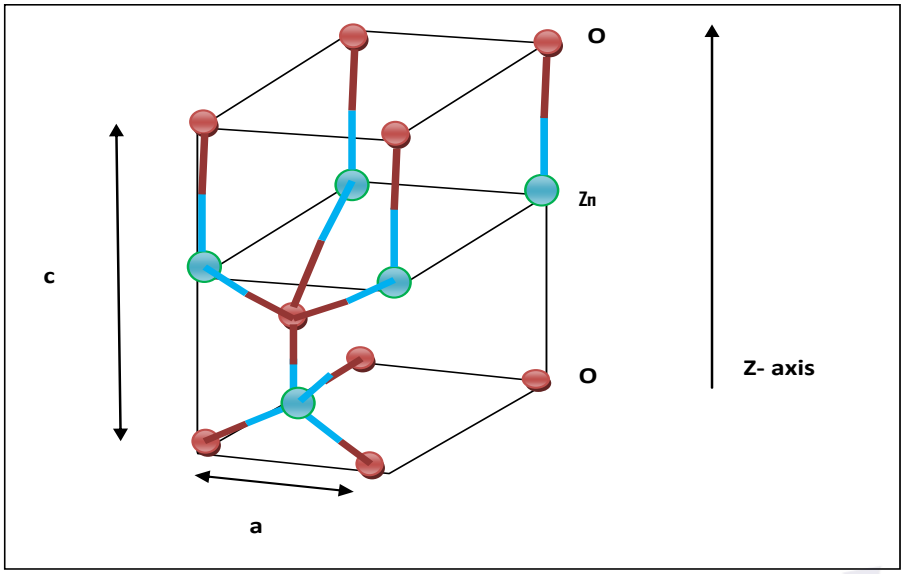

Figure 1.3: The hexagonal wurtzite of ZnO crystal structure

The wurtzite structure of ZnO consists four common surfaces; two of them are polar i.e Zn (001) and O (00-1) whose faces are terminated along c-axis and two are non polar i.e (11-20) and (10-10) faces. The non polar surfaces possess an equal number of Zn and O atoms (Ali, 2012). The primitive translation vectors a= 0.3249 nm and b=0.3249 nm lay in the X-Y plane are of equal length and include angle of 120° while c=0.5206 nm is parallel to z-axis. The other possible lattice parameters of the hexagonal unit cell obtained in various methods range from 0.32475-0.32501 nm for " a" and from 0.52042-0.52075 nm for " c" (at room temperature) (Ramashia, 2015). ZnO has a high melting point 1975 °C which determine its high thermal and chemical stability. The other properties of ZnO (bulk) are given in table 1.3. The conduction band minima and valance band maxima appear at the same axis Brillouin zone, indicating that ZnO is direct semiconducting material (Tobin, 2005). The direct and wide band gap properties also suggest that ZnO may have a high breakdown voltage and may have lower noise which can be used in high-temperature and high-power applications. The number of articles published on ZnO has been increasing every year and in 2007-2008 and ZnO becomes the second popular semiconductor after Si due to improvements in growth related techniques of single crystalline ZnO.

Table 1.3: Physical properties of bulk ZnO

Property	Value
Density	5.67526 g/cm^3
Molecular mass	81.389
Point group	6 mm (Wurtzite)
Lattice constants at room temp.	a=3.250Å, c=5.205Å
Melting point	2250K
Electron mass	0.28
Hole mass	1.8
Band gap energy at room temperature	3.37 eV
Exciton binding energy	60 meV
Specific heat	0.125 cal/gm
Thermal conductivity	0.006 cal/cm/K
Thermoelectric constant at 573 K	1200 mV/K

1.3 Nanostructured Zinc Oxide

Zinc oxide is a compound semiconductor which consists of Zn+2 and O-2. Zinc belongs to group II of transition metals and oxygen to group VI element; therefore, it is called a metal oxide semiconductor. The nanostructured materials have been broadly studied due to their potential uses in fabricated micro and nanoscale devices. Since the discovery of carbon nanotubes in 1911 many experimental and theoretical investigations have been conducted on nanoscale ZnO such as nanowires, nanorods and nanotubes (Iijima, 1991). Nanostructured ZnO has a large surface to volume ratio that can be used to enhance the performance of devices. As the sizes are reduced, devices become faster. The reduction in size also contributes to the quantum confinement phenomenon. Quantum confinement traps electrons in a small area in such a way that controlling the moment of electron in particular direction becomes easier. Confinement of electrons within the small-sized material also renders the band gap tunable; additionally, confinement increases the optical bandgap and promotes a faster operation speed (Lu et al., 2006). Nanostructured ZnO has achieved particular consideration due to its distinguished performance in photonics, optics and electronics, ZnO is the richest nanostructured material, i.e can be formed in nanocomb, nanorings, nanospring/nanohelix, nanobelt, nanowires and nanocages form (Wang, 2004).

ZnO is a unique material and it is extensively researched by scientists for its potential in a wide range application such as dielectric, piezoelectric, pyroelectric, plate panel display sensor devices, field effect transistors and ultraviolet light emitting devices (Kim., 2011). In nanostructured form ZnO has researched extensively over the past few decades due to their exclusive properties compared to its bulk form. Nanostructured ZnO consists of the structures with diameter not more than 100nm (Pei et al., 2009). A lot of nanostructures were produced by scientists; however, it cannot be determined that which nanostructure is best for electronic, optoelectronic device applications (Buhro & Colvin, 2003). From 1960, synthesis of ZnO thin film has been an active field because of their applications as sensors, transducers and catalysts. In Figure 1.4 the research studies available over the previous year's regarding ZnO nanomaterial indicates that among the other nanomaterials ZnO is the most attractive and its applications in chemical/gas sensors is substantially increased (Galstyan, et al., 2016).

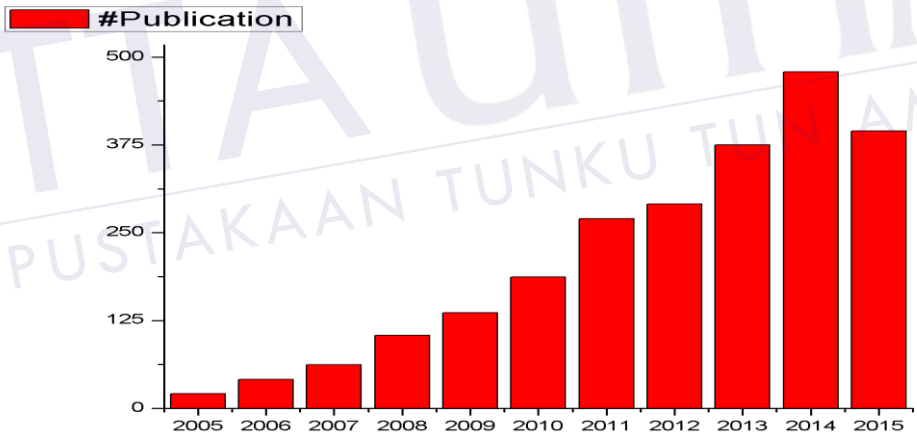

Figure 1.4: Number of publications on ZnO nano and gas or chemical sensor according to web of knowledge database

The distinctive properties of 1-D ZnO nanostructures grant a promising combination for chemical selectivity electrochemically and chemically tunable platform is crucial for tailor sensor response. For that reason, 1-D ZnO nanostructure NRs/NWs are comprehensively applied in a variety of sensing applications e.g biomarkers, biosensors, chemical sensors, gas sensors, humidity sensors, pH sensors pressure/force/load sensors and UV sensors. In the last few decades nanotechnology

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